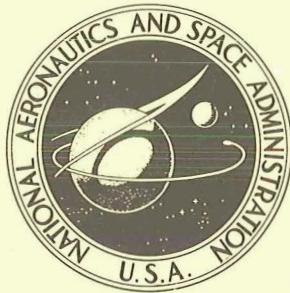


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PREPARATION AND PROPERTIES OF IMIDE/PYRRONE COPOLYMERS

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SUMMARY

Two series of copolymers containing imidazopyrrolone (Pyrrone) and imide groups were prepared by solution polymerization. The mechanical properties of thin films of the copolymers were measured and the films showed a general increase in the tangent modulus and a decrease in elongation with increasing Pyrrone content. The copolymers were more resistant to degradation by strong acids and bases than the corresponding polyimide. The thermal stabilities of the copolymers in air improved with increasing imide content, while the thermal stabilities in a vacuum improved with increasing Pyrrone content. These copolymers represent a way to utilize the useful properties of each class of homopolymers.

INTRODUCTION

It has become apparent during the past decade that the conventional plastics developed during the first half of the 20th century have been unable to meet the more demanding needs of aeronautical and aerospace research and technology. However, two new classes of polymers, the polyimides (refs. 1 and 2) and the polyimidazopyrrolones (Pyrrones) (refs. 3 to 7), have been developed recently and show promise of meeting these critical materials needs. These new polymers derive their unique properties from the incorporation of a combination of aromatic and heterocyclic structural units, with their high orders of chemical, thermal, and radiation stability, into the long molecular chains that are the essentials of polymers. As the first commercially available heteroaromatic polymer to be offered in a variety of fabricated forms, the polyimides are currently being evaluated and even utilized in numerous aeronautical applications. The Pyrrones, a class of heteroaromatic polymers discovered at the Langley Research Center, represent an extension of the scheme of fusing aromatic and heterocyclic structural units into polymers of the ladder or two-strand variety that begin to approach the general structure of graphitic materials.

Though close similarities in the properties of the two polymer classes exist, there are also features unique to each class. The polyimides are considered to be essentially linear because of their solubilities in fuming nitric acid (ref. 2). However, the Pyrrones,

which are prepared by virtually the same solution polymerization technique as the polyimides, are virtually insoluble in strong acidic solvents (ref. 7); this insolubility suggests a certain element of nonlinearity by reason of cross-linked sites. Compared with the polyimide films, the Pyrrone films exhibit high tangent moduli and low elongations, which are indicative of greater rigidity of the polymer chains. Thermogravimetric behavior of the two classes in a vacuum also shows a difference, the Pyrrones demonstrating lower weight losses in the extreme temperature range of 873° to 1173° K (600° to 900° C).

Either or both of these two polymer classes could quite possibly be used in specific aerospace or aeronautical applications. However, it was thought that by combining the two polymer types by copolymerization, a series of copolymers might be obtained that would have a range of properties. The resulting versatility can be imagined; for example, consider the properties that a polymer film must have in order to perform as a cell separator in an alkaline battery. For this end use, a Pyrrone film would probably resist the chemical action of the caustic electrolyte yet would likely be too brittle to serve as an unsupported membrane. Even though the polyimides are quite flexible as films, they are known to be dissolved by caustics. The solution could be to employ a copolymer of Pyrrone and polyimide if the two desirable properties of each class, resistance to alkali and film flexibility, could be merged into the copolymer.

In order to establish the scope of the properties that could be realized by copolymerization, two chemically differing series of copolymers were prepared covering the range of composition from a pure Pyrrone to a pure polyimide with various intermediate copolymer compositions. The copolymers were then studied from the standpoint of the solution properties of the intermediate prepolymers, the chemical resistance of the cured films, and the mechanical properties of the films.

SYMBOLS AND ABBREVIATIONS

BTDA 3,3',4,4'-benzophenone tetracarboxylic dianhydride

DAB 3,3'-diaminobenzidine

DMAc N,N-dimethylacetamide

N normality, equivalent weights/decimeter³

n degree of polymerization

ODA p,p'-oxydianiline

PMDA	pyromellitic dianhydride
R.H.	relative humidity
TADPO	3,3',4,4'-tetraaminodiphenyl ether
η_{int}	intrinsic viscosity, deciliters/gram

EXPERIMENTAL PROCEDURES

The schematic representations for the reactions leading to both Pyrrone polymers and polyimides are given in equations (A1) and (A2), respectively, in appendix A. The two copolymer series were prepared by the reactions illustrated by equations (A3) and (A4) in appendix A. Pyromellitic dianhydride (PMDA), p,p'-oxydianiline (ODA), and 3,3',4,4'-tetraaminodiphenyl ether (TADPO) were used as the starting materials for the series of copolymers that were prepared by the reaction shown in equation (A3). This series of copolymers is referred to in this report as the PMDA-ODA/TADPO series. The second group of copolymers, which is represented by equation (A4), was prepared by utilizing 3,3',4,4'-benzophenone tetracarboxylic dianhydride (BTDA), 3,3'-diaminobenzidine (DAB), and ODA as the basic chemicals. This series is referred to as the BTDA-ODA/DAB series. The numerical ratios appearing in equations (A3) and (A4) indicate the relative amounts of diamines (ODA) and tetraamines (TADPO and DAB) used to prepare the copolymers.

Polymerizations

A summary of the polymerizations leading to the PMDA-ODA/TADPO copolymers is given in table I, and a summary of the polymerizations for the BTDA-ODA/DAB copolymers is listed in table II. The purification of the various monomers is given in appendix B. Detailed procedures for the synthesis of the copolymers are given in the following sections.

PMDA-ODA/TADPO (100/0).- The PMDA-ODA/TADPO (100/0) control polyimide was prepared by the addition of 4.00 grams of solid PMDA to a stirred solution (high-speed household blender) of 4.00 grams of ODA in 60 cm³ of N,N-dimethylacetamide (DMAc). After 15 minutes, all the PMDA had dissolved and the temperature of the solution had increased to between 308° and 313° K (35° and 40° C). A solution of 0.55 gram of PMDA in 14 cm³ of DMAc was added dropwise to the stirred polymer solution until the desired consistency was obtained. The measured viscosity of the resulting prepolymer is presented in table I.

PMDA-ODA/TADPO (90/10, 75/25, 50/50, 25/75, 0/100). - The PMDA-ODA/TADPO copolymers (90/10, 75/25, 50/50, 25/75) and the PMDA-ODA/TADPO (0/100) control Pyrrone were prepared by the following procedure, which is illustrated for the 90/10 copolymer. A solution of 4.00 grams of PMDA in 30 cm³ of DMAc was added to a rapidly stirred solution of 3.60 grams of ODA and 0.46 gram of TADPO in 35 cm³ of DMAc. The reaction resulted in heating of the polymer solution to between 308° and 313° K (35° and 40° C). After the resulting solution had been stirred for 15 minutes, a solution of 0.55 gram of PMDA in 10 cm³ of DMAc was added dropwise. This total addition of 4.55 grams of PMDA resulted in a viscous polymer solution $\eta_{int} = 1.65 \text{ dl/g}$ in DMAc at 298° K (25° C).

BTDA-ODA/DAB (100/0, 75/25, 50/50, 25/75, 0/100). - The BTDA-ODA/DAB (100/0) control polyimide was synthesized by the same method (addition of solid dianhydride) as that used to prepare the PMDA-ODA/TADPO (100/0) control polyimide, and the BTDA-ODA/DAB copolymers (75/25, 50/50, 25/75) and the BTDA-ODA/DAB (0/100) control Pyrrone were prepared by the same method (addition of dianhydride solution) as that used for preparation of the corresponding PMDA-derived copolymers.

Film Preparation

Films were obtained by solvent-casting the previously described polymer solutions onto glass plates, drying them at 373° K (100° C) for 2 hours, and curing them at 448° K (175° C) for 1 hour and then at 573° K (300° C) for 1 additional hour. The resulting films were easily stripped from the glass plates and used for testing. The copolymer films were light yellow for the polyimides and progressed through orange shades with increasing Pyrrone content to the deep red color of the pure Pyrrone. The infrared spectra of the PMDA-ODA/TADPO copolymers, which were obtained in order to confirm the composition, appeared to be composites of the spectra of the two homopolymers: PMDA-ODA polyimide (ref. 2) and PMDA-TADPO Pyrrone (ref. 3). The intensity of the absorption bands at 5.63 μ , 9.85 μ , and 16.6 μ in the copolymer spectra increased with increasing imide (ODA) content, while the intensity of the bands at 6.19 μ and 10.4 μ increased with increasing Pyrrone (TADPO) content.

RESULTS AND DISCUSSION

Mechanical Properties

The mechanical properties of the films derived from the two series of copolymers were determined by the technique described in appendix C. They are listed in tables III and IV and are pictured in figures 1 and 2. The films varied in thickness from about 0.80 to 1.30 mils (0.02 to 0.03 mm), although the individual test specimens for each film composition were the same thickness to within 0.10 mil (0.0025 mm).

The properties of both series of films reflect the changes to be expected for a progressive stiffening of the polymer chains (increased film brittleness) with increasing Pyrrone (TADPO and DAB) content. The tangent moduli show a general increase and the elongations show a corresponding decrease. Although less consistent, the yield and tensile strengths of the two series of films increase with increasing Pyrrone content.

Thermal Stability

The thermal stabilities of the two copolymer series were determined by heating the films in a vacuum using the thermogravimetric analytical procedure. The results are shown in figure 3 for the PMDA-ODA/TADPO films and in figure 4 for the BTDA-ODA/DAB films. The general effect of copolymerization was an increase in the thermal stability over that of the polyimides, since for both series, the char yields at 1073° K (800° C) were greater for the copolymers containing more Pyrrone structure (higher percentages of TADPO and DAB).

The thermal stabilities of the copolymers were also measured by thermogravimetric analysis in an environment of air. The relative oxidative stabilities are pictured in figure 5 for the PMDA-ODA/TADPO films and in figure 6 for the BTDA-ODA/DAB films. The thermal stability of the PMDA-derived series increases slightly with increasing imide content. Because of the close similarities of the thermogravimetric curves obtained for the BTDA-derived copolymers, they have all been represented as falling within the shaded area outlined by the curves for the BTDA-ODA/DAB (100/0) control polyimide and the BTDA-ODA/DAB (0/100) control Pyrrone films. For a comparison of the two series, the curve for the control polyimide of the PMDA-derived series has been indicated by a dashed line.

The effect of copolymerization on the oxidative stability of the PMDA-ODA/TADPO copolymer series is pictured in a different manner in figure 7. Here, the temperatures of maximum rates of decomposition of the copolymers, as determined by differential thermal analysis, are plotted as a function of Pyrrone/imide (TADPO/ODA) content, and it is apparent that the temperatures of the maximum rates of decomposition for the copolymers increase with increasing imide content. A corresponding evaluation of the BTDA-ODA/DAB series was not made.

Thus, it can be concluded that even though the thermal stabilities of the imide/Pyrrone copolymers in a vacuum environment are greater with increasing Pyrrone content, the effect of copolymerization on the oxidative stabilities is quite the opposite; that is, the copolymers with the higher imide content are superior in a thermal air environment.

Chemical Stability

The resistance of the imide/Pyrrone copolymers to strong caustic and acidic reagents was more similar to that of the pure Pyrrones (ref. 7) than to that of the polyimides (ref. 2). As little as 10 percent of the Pyrrone structure resulted in a marked change in chemical resistance, as shown in table V for the PMDA-ODA/TADPO series. The reason for such an enhancement of the chemical resistance by the inclusion of Pyrrone structures has not been determined with certainty. The likelihood that cross-linking is the main factor is strong. However, as little as 10 percent Pyrrone structure (PMDA-ODA/TADPO (90/10)) results in a marked change in insolubility while the mechanical properties of the film are not markedly changed from those of the pure polyimide film (PMDA-ODA/TADPO (100/0)), as would be expected from a large amount of cross-linking; therefore, some doubt is cast on the validity of the cross-linking explanation.

An experiment that not only emphasized the chemical stability of the imide/Pyrrone copolymers, but also substantiated their copolymeric nature involved soaking 1-mil (0.0254-mm) films of the PMDA-ODA/TADPO (75/25, 25/75) copolymers in yellow fuming nitric acid. After 24 hours, the 75/25 and 25/75 copolymers had lost only 5 percent and 2 percent of their weights, respectively. In contrast, a film prepared from a simple mixture of 75 percent PMDA-ODA polyimide and 25 percent PMDA-TADPO Pyrrone prepolymers lost 57 percent of its weight after identical treatment. This result proves that the copolymers were not mere physical mixtures.

Water Absorption

The regular variation of the properties of the copolymers with respect to their constituent groups (Pyrrone and imide) was further exemplified by the amounts of water absorbed readily by thin films of the two copolymer series. The weight percent of water absorbed at 298° K (25° C) and 100 percent relative humidity are plotted as a function of the amount of Pyrrone content (TADPO and DAB) for the PMDA-ODA/TADPO series in figure 8 and for the BTDA-ODA/DAB series in figure 9. Though the increased absorptivity of water caused by copolymerization of the polyimides with the Pyrrones would be less than desirable for such end uses as electrical insulation, there can also be imagined uses wherein the hydrophilic nature would be desirable, as in membranous end uses.

CONCLUDING REMARKS

It has been demonstrated that polyimides and Pyrrone polymers can be copolymerized to give materials that have a potential for use in aeronautical and aerospace applications. The characteristics of the resultant copolymers can be varied to give a versatile

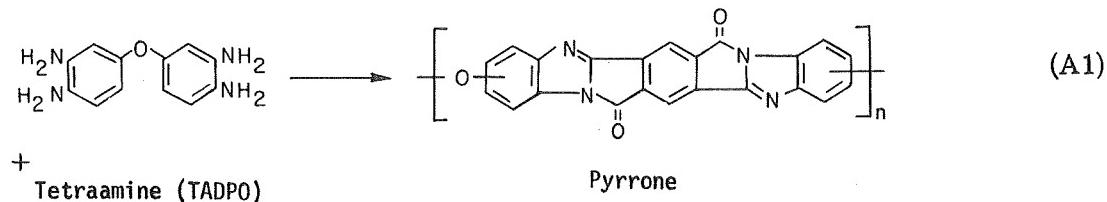
selection of properties. This variation in properties is generally regular in dependence upon the compositions of the copolymers between the extremes of the pure polyimides and the pure Pyrrones. An example of the value of this variability in properties is shown by the moderation of the inherent brittleness of Pyrrone films by the incorporation of certain quantities of imide groups into the polymer by copolymerization.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Station, Hampton, Va., October 1, 1969.

APPENDIX A

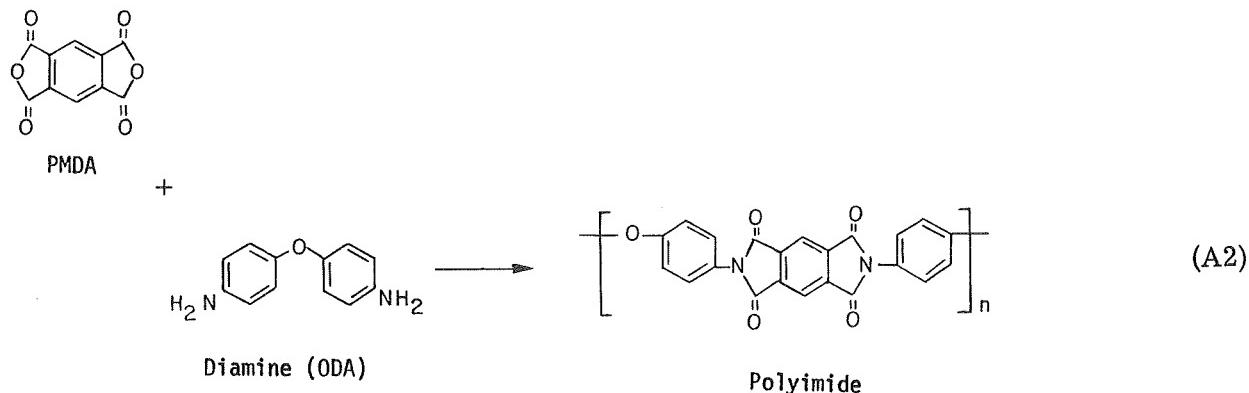
ANALYSIS OF CHEMICAL REACTIONS

The schematic reactions of aromatic dianhydrides (for example, PMDA) with aromatic tetraamines (for example, TADPO) and aromatic diamines (for example, ODA) leading to Pyrrones and polyimides are depicted in equations (A1) and (A2), respectively.



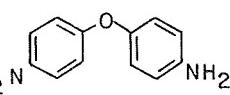
+
Tetraamine (TADPO)

Pyrrole



PMDA

+

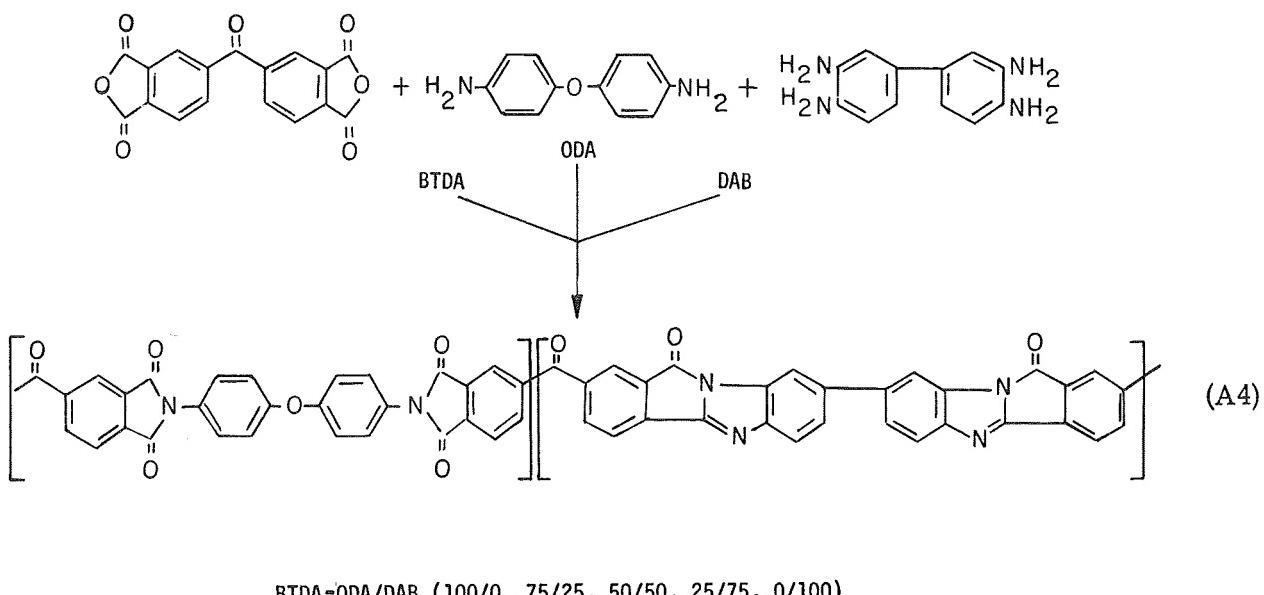
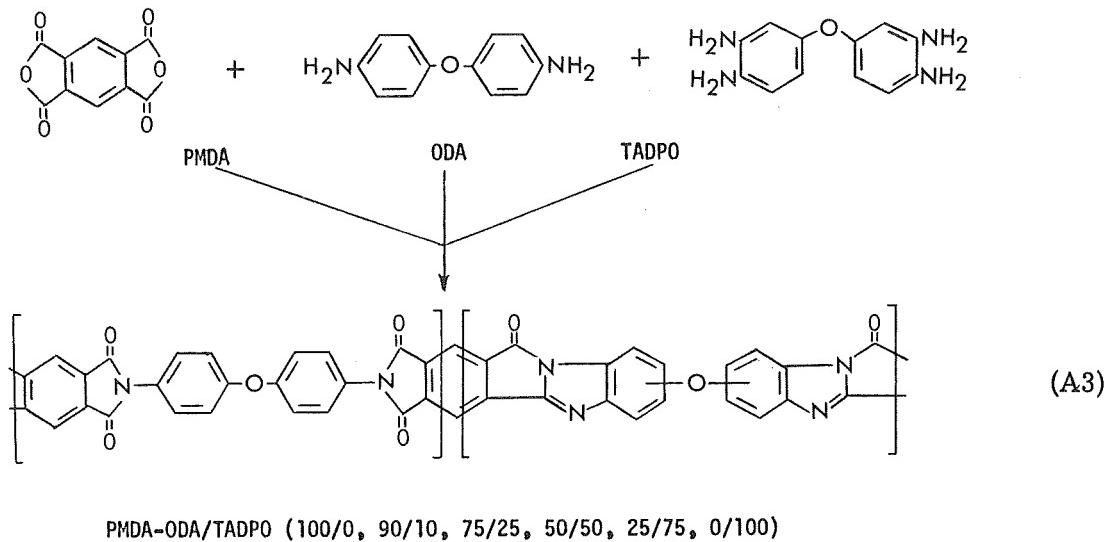


Diamine (ODA)

Polyimide

The overall reaction for the PMDA-derived copolymer series is given in equation (A3) and the overall reaction for the BTDA-derived series is illustrated in equation (A4).

APPENDIX A – Concluded



APPENDIX B

PURIFICATION OF MONOMERS

Pyromellitic dianhydride was purified by vacuum sublimation at 498° to 513° K (225° to 240° C) and 0.5 torr (66.5 N/m^2). $3,3',4,4'$ -Benzophenone tetracarboxylic dianhydride was purified by recrystallization from acetic anhydride. $3,3',4,4'$ -Tetraaminodiphenyl ether was synthesized and purified by a method described in references 7 and 11. p,p' -Oxydianiline was purified by recrystallization from acetone, followed by sublimation at 458° K (185° C) and 0.5 torr (66.5 N/m^2). $3,3'$ -Diaminobenzidine was used as received from commercial sources. N,N -Dimethylacetamide was purified by distillation from pyromellitic dianhydride.

APPENDIX C

MEASUREMENT OF PROPERTIES

Viscosity measurements were performed with Ubbelohde viscometers at 298° K (25° C) in DMAc. The intrinsic viscosity was obtained from a four-point extrapolation to infinite dilution (ref. 8). Thermogravimetric analyses were performed by using a null-balancing automatic-recording electrobalance (Cahn RG electrobalance) with 1-mil (0.0254-mm) film samples weighing 2.05 ± 0.03 mg (ref. 9). The vacuum measurements were made at a pressure of 1×10^{-6} torr (1.33×10^{-4} N/m²) maintained by an ion pump with a capacity of 250 000 cm³/sec. Differential thermal analyses were made by using a Du Pont 900 Differential Thermal Analyzer (ref. 10).

The tensile stress-strain tests were performed with a Model TTC Instron Universal Tester on at least 10 separate 0.5-inch-wide (1.27-cm) strips of film by utilizing a 3.0-inch (7.62-cm) grip separation and a crosshead speed of 0.2 inch/min (0.51 cm/min). From the stress-strain curve, it was possible to determine the initial tangent modulus, the 0.2-percent offset-yield strength, and the tensile strength and elongation at break.

The moisture absorption was determined by exposing 2.0-mg samples of 1-mil (0.0254-mm) film to alternate environments of 100 percent relative humidity at 298° K (25° C) and zero percent relative humidity at 373° K (100° C). The following equation was used:

$$\text{Percent of water} = \frac{(\text{Weight at } 100\% \text{ R.H., } 298^{\circ} \text{ K}) - (\text{Weight at } 0\% \text{ R.H., } 373^{\circ} \text{ K})}{\text{Weight at } 0\% \text{ R.H., } 373^{\circ} \text{ K}} \times 100$$

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TABLE I.- SUMMARY OF POLYMERIZATIONS FOR PMDA-ODA/TADPO COPOLYMERS

ODA/TADPO ratio	Reactants			Solvent (DMAc), cm ³	Intrinsic viscosity, dl/g
	ODA, millimoles	TADPO, millimoles	PMDA, millimoles		
100/0	20.0	-----	20.8	74	1.47
90/10	18.0	2.0	20.8	75	1.65
75/25	15.0	5.0	20.8	75	2.55
50/50	10.0	10.0	20.6	75	2.78
25/75	5.0	15.0	21.1	75	1.08
0/100	-----	20.0	21.0	75	.99

TABLE II.- SUMMARY OF POLYMERIZATIONS FOR BTDA-ODA/DAB COPOLYMERS

ODA/DAB ratio	Reactants			Solvent (DMAc), cm ³	Intrinsic viscosity, dl/g
	ODA, millimoles	DAB, millimoles	BTDA, millimoles		
100/0	20.0	-----	20.5	60	1.05
75/25	15.0	5.0	20.2	60	1.18
50/50	10.0	10.0	20.2	60	1.28
25/75	5.0	15.0	20.1	60	.85
0/100	-----	20.0	20.1	60	.83

TABLE III.- MECHANICAL PROPERTIES OF PMDA-ODA/TADPO COPOLYMER FILMS

Copolymer composition	Tangent modulus		Elongation, percent	Strength			
				Yield		Tensile	
	ksi	N/m ²		ksi	N/m ²	ksi	N/m ²
PMDA-ODA/TADPO (100/0)	344	237×10^7	7.7	4.6	3.2×10^7	11.3	7.8×10^7
PMDA-ODA/TADPO (90/10)	405	279	7.9	6.1	4.2	13.9	9.6
PMDA-ODA/TADPO (75/25)	502	346	4.7	8.6	5.9	14.7	10.1
PMDA-ODA/TADPO (50/50)	415	286	4.3	8.8	6.1	14.1	9.7
PMDA-ODA/TADPO (25/75)	589	406	3.4	10.7	7.4	14.0	9.6
PMDA-ODA/TADPO (0/100)	623	429	3.1	11.4	7.9	14.3	9.9

TABLE IV.- MECHANICAL PROPERTIES OF BTDA-ODA/DAB COPOLYMER FILMS

Copolymer composition	Tangent modulus		Elongation, percent	Strength				
				Yield		Tensile		
	ksi	N/m ²		ksi	N/m ²	ksi	N/m ²	
BTDA-ODA/DAB (100/0)	376	259×10^7	5.2	8.7	6.0×10^7	12.4	8.5×10^7	
BTDA-ODA/DAB (75/25)	444	306	4.7	10.3	7.1	14.7	10.1	
BTDA-ODA/DAB (50/50)	443	305	5.0	9.7	6.7	14.2	9.8	
BTDA-ODA/DAB (25/75)	569	392	3.5	13.3	9.2	16.0	11.0	
BTDA-ODA/DAB (0/100)	588	405	3.7	12.9	8.9	17.3	11.9	

TABLE V.- EFFECT OF CAUSTIC AND ACIDIC REAGENTS ON
PMDA-ODA/TADPO COPOLYMER FILMS^a

Copolymer composition	2N NaOH	96% H ₂ SO ₄	Fuming HNO ₃
PMDA-ODA/TADPO (100/0)	Gelled mass in 0.5 hr	Gelled mass in 1.0 hr	Dissolved in 0.5 hr
PMDA-ODA/TADPO (90/10)	Film intact for over 48 hr	Film intact for over 48 hr	Dissolved in 24 to 48 hr
PMDA-ODA/TADPO (75/25, 50/50, 25/75)			Film intact for over 48 hr
PMDA-ODA/TADPO (0/100)			

^aTests performed on 1-mil (0.0254-mm) films at room temperature.

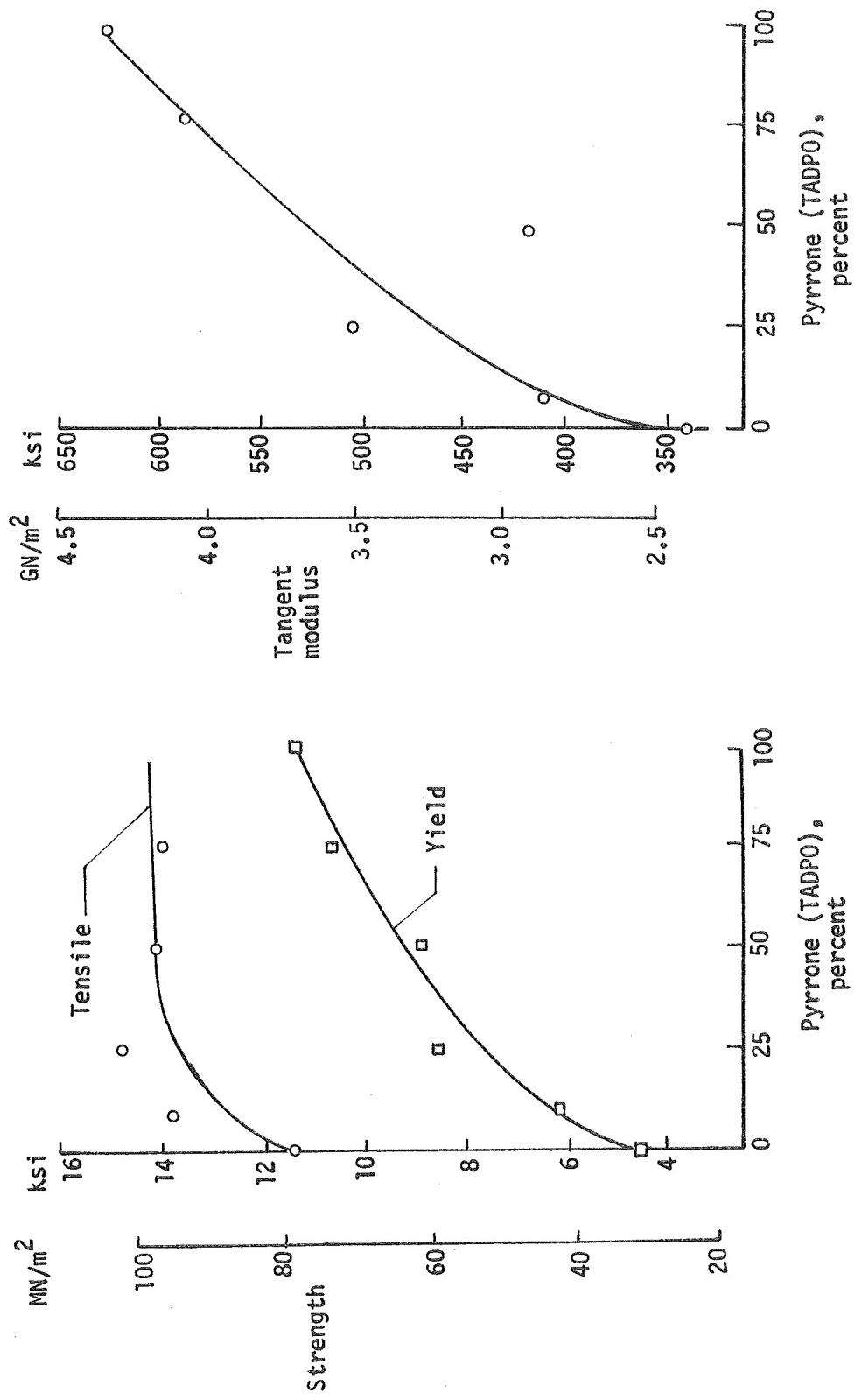


Figure 1.- Tensile properties of PMDA-ODA/TADPO copolymer films.

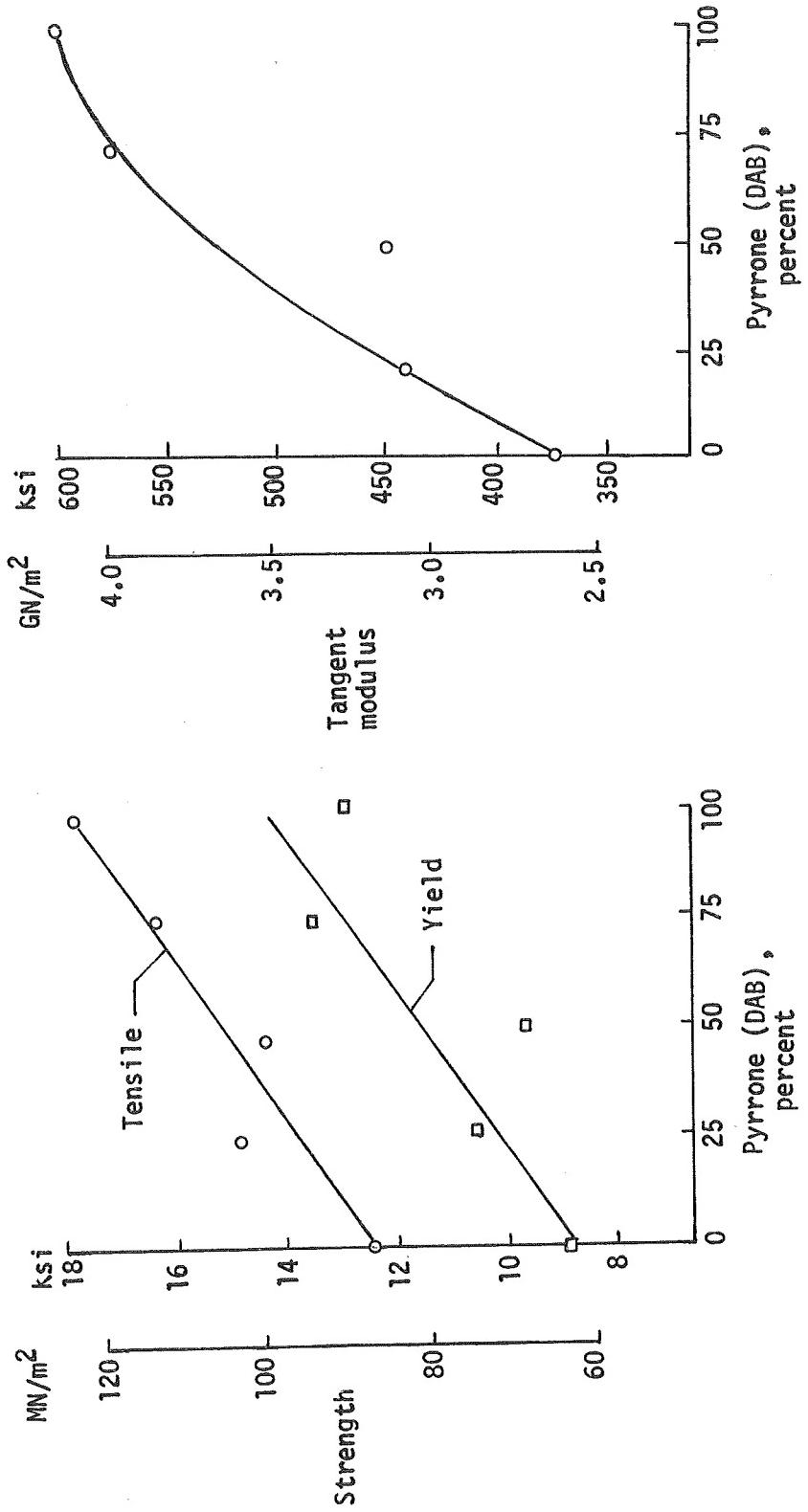


Figure 2.- Tensile properties of BTDA-ODA/DAB copolymer films.

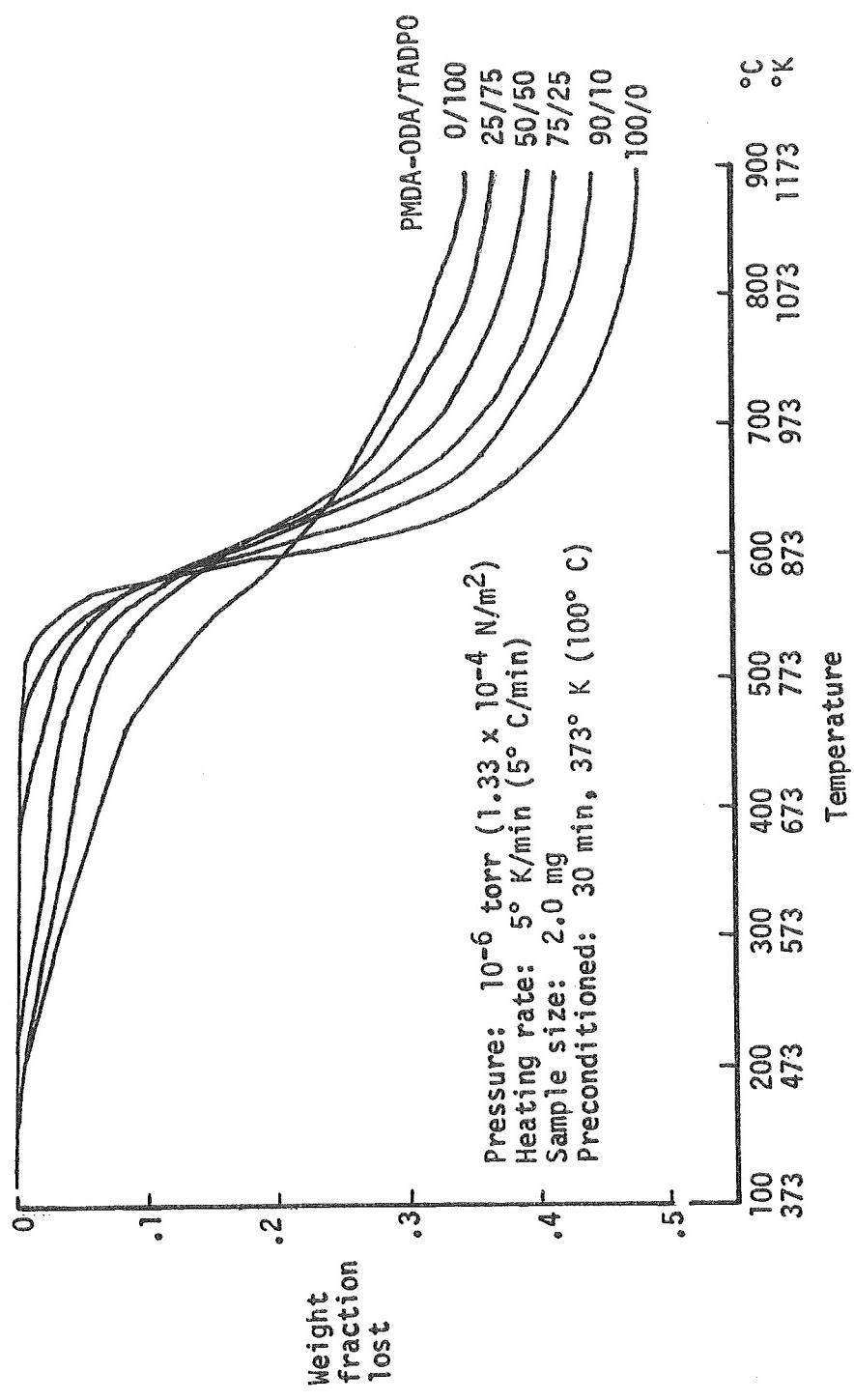


Figure 3.- Thermogravimetric analyses of PMDA-ODA/TADPO copolymer films in vacuum.

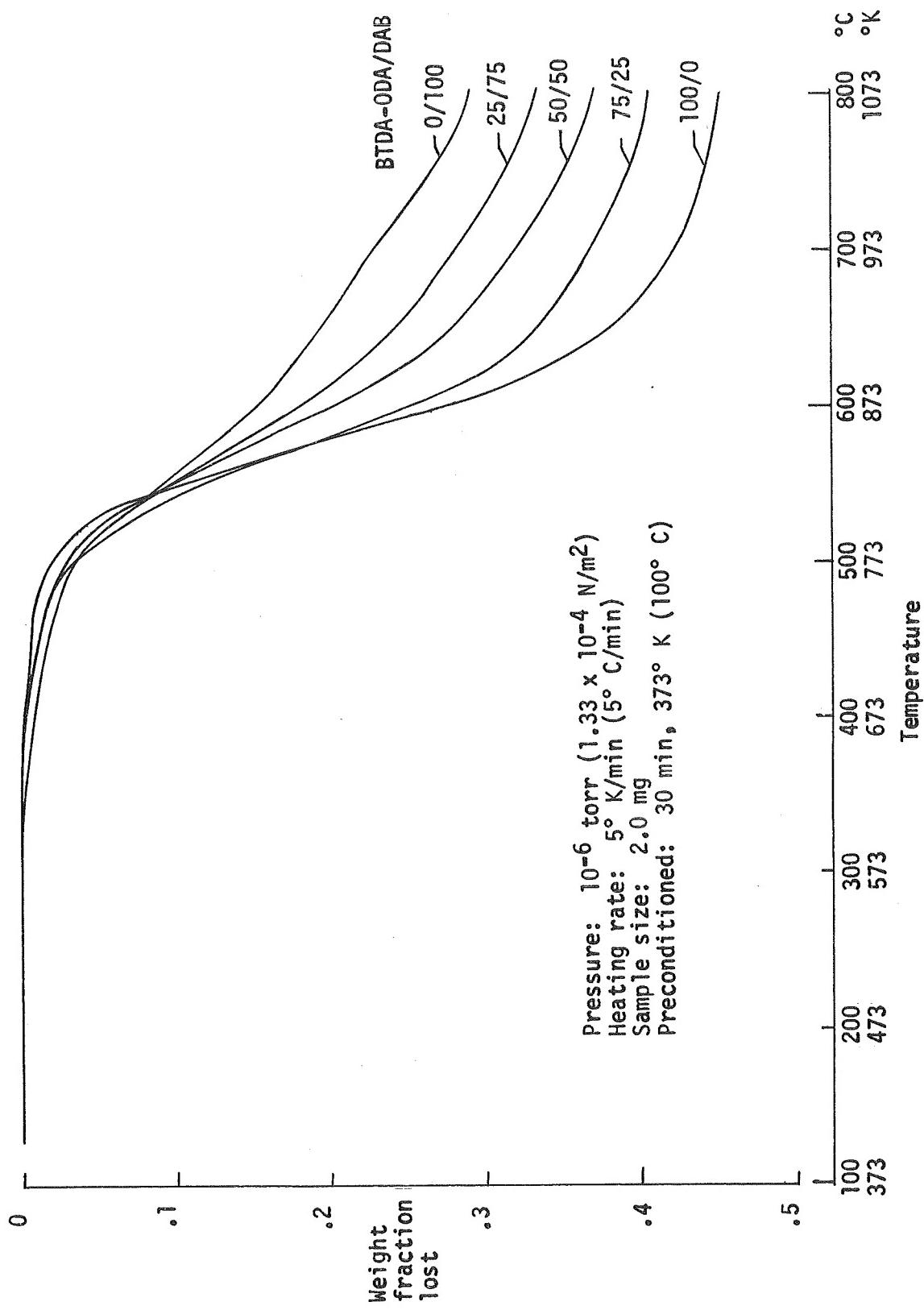


Figure 4.- Thermogravimetric analyses of BTDA-ODA/DAB copolymer films in vacuum.

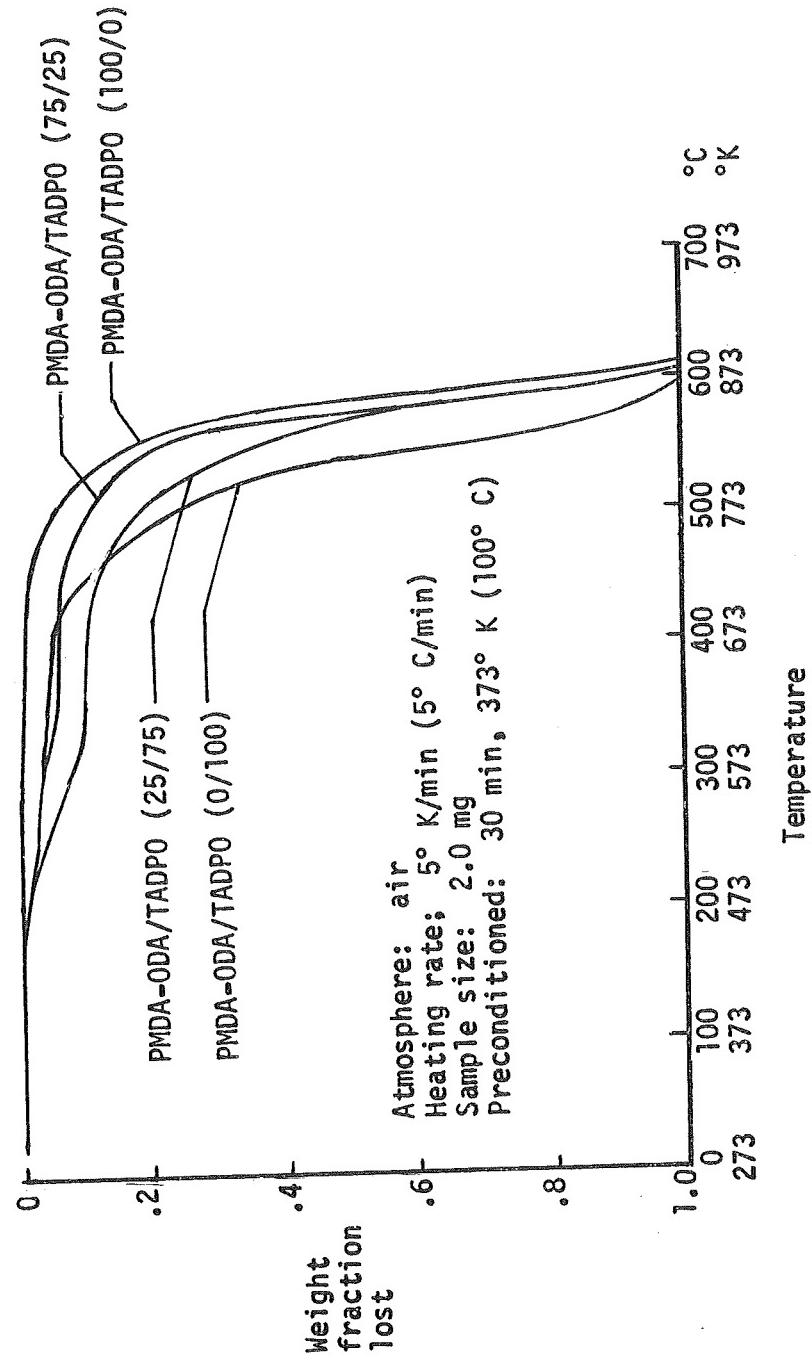


Figure 5.- Thermogravimetric analyses of PMDA-ODA/TADPO copolymer films in air.

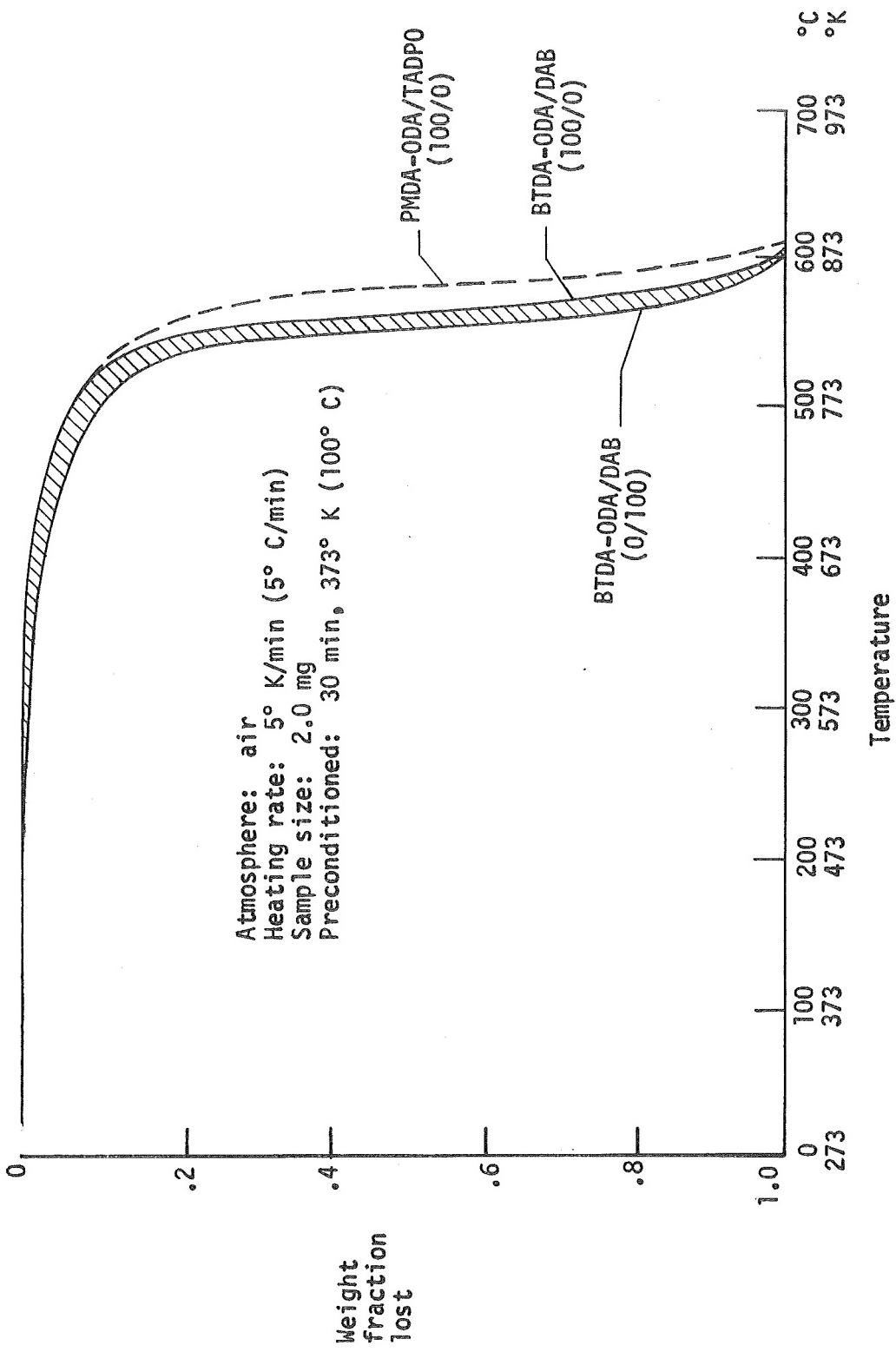


Figure 6.- Thermogravimetric analyses of BTDA-ODA/DAB copolymer films in air.

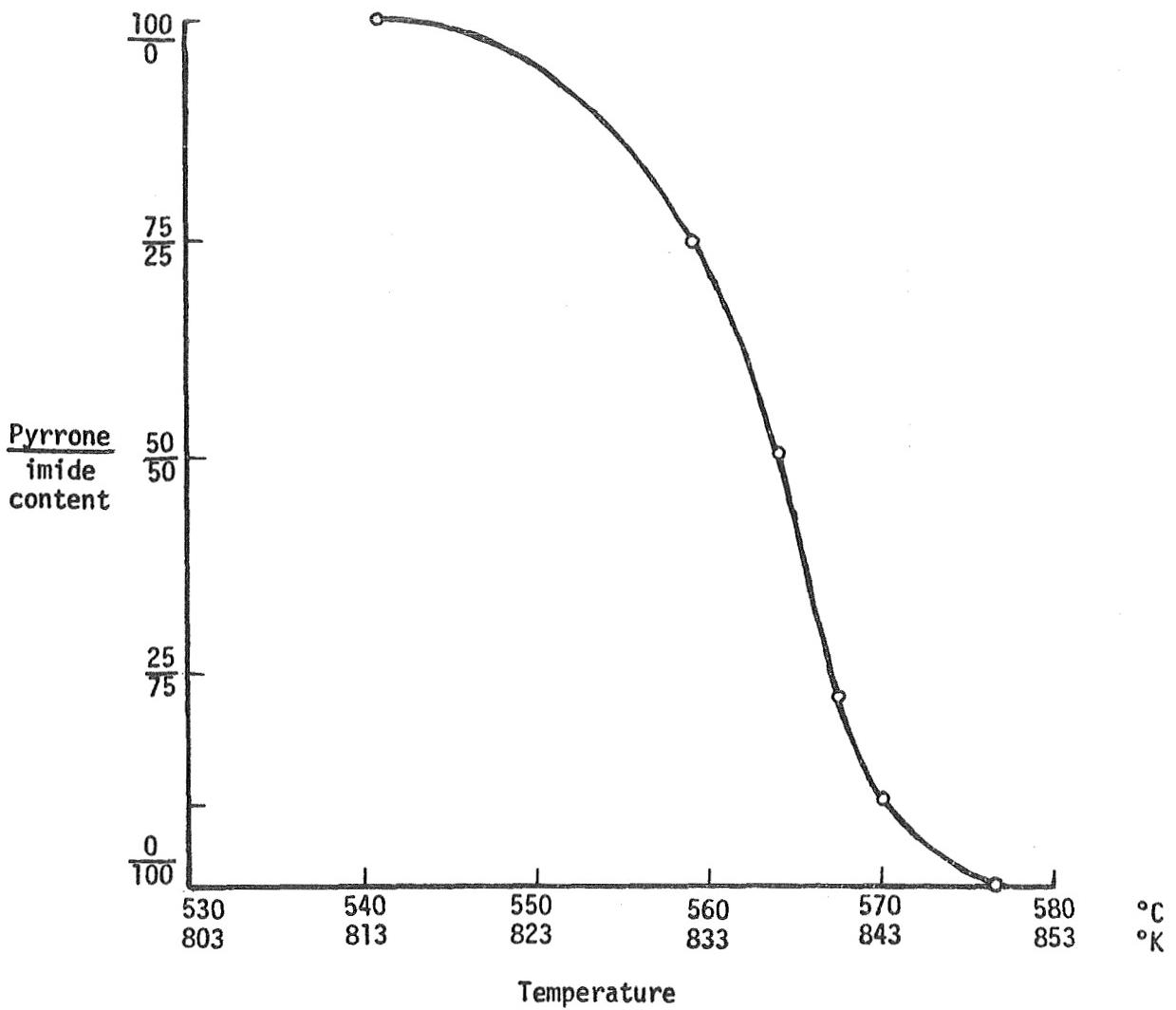


Figure 7.- Temperatures of maximum rates of decomposition in air of PMDA-ODA/TADPO copolymer films as a function of Pyrrone/imide content.

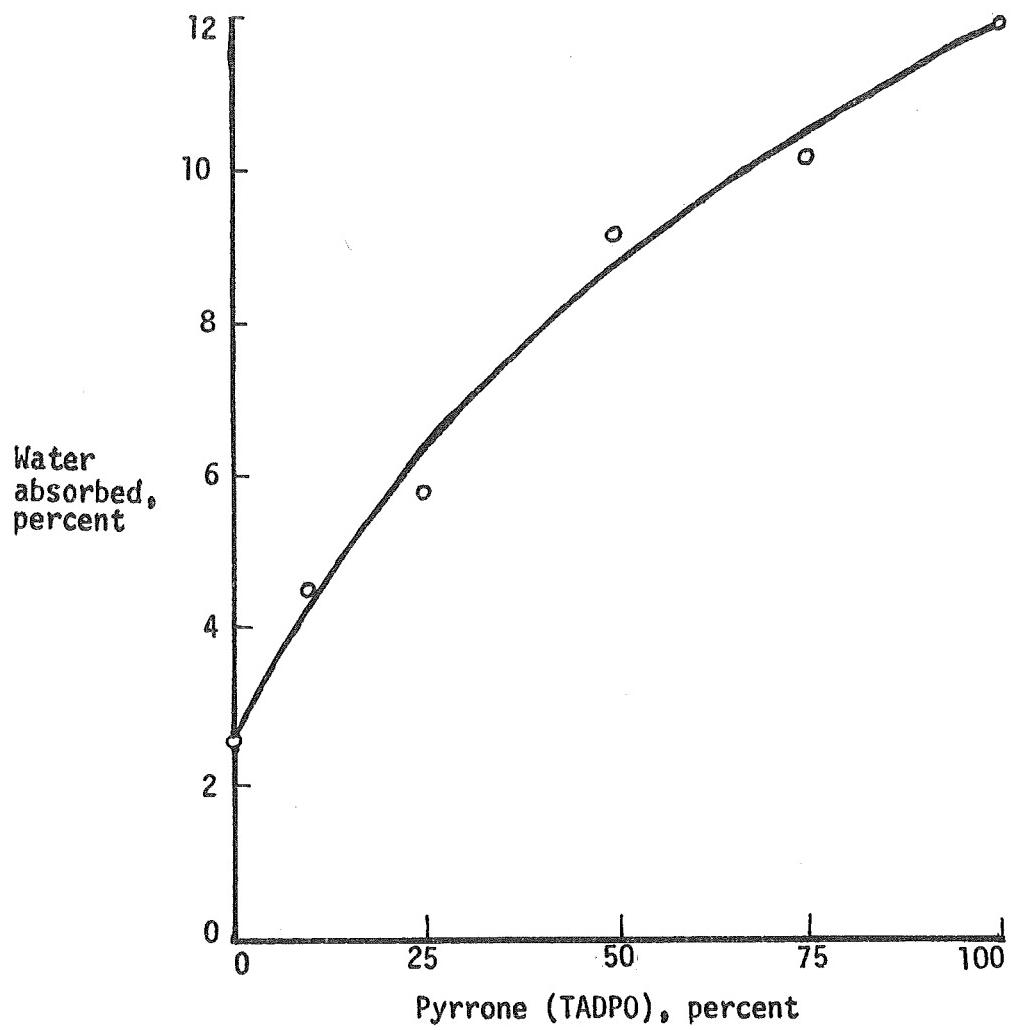


Figure 8.- Percentage water absorption by PMDA-ODA/TADPO copolymer films.

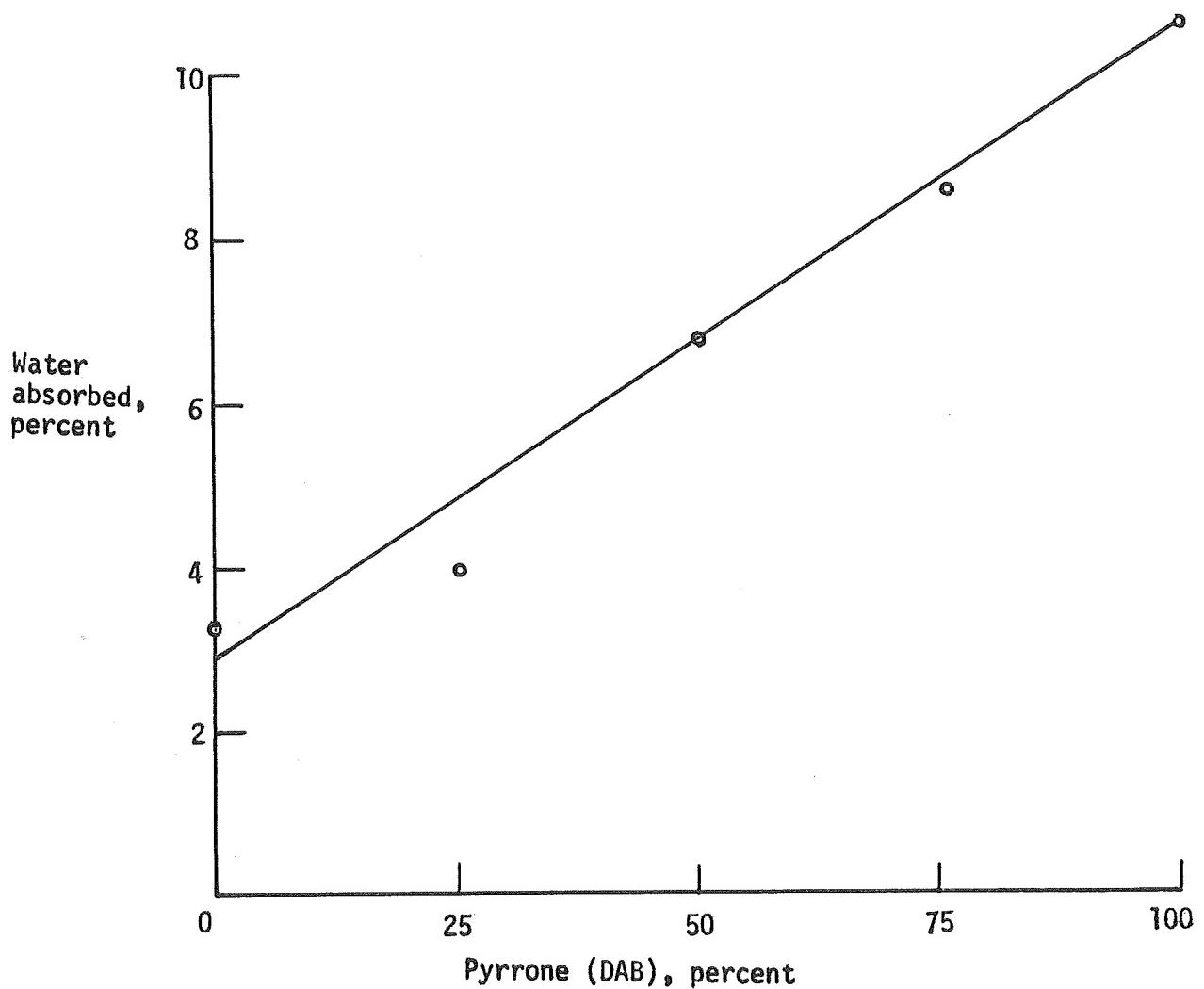


Figure 9.- Percentage water absorption by BTDA-ODA/DAB copolymer films.

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